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(57) Abstract

The invention relates to foodstuffs containing improved sucrose-like tasting sweetener compositions with aspartame as the principal source of sweetness and another compound delivering sweetness, wherein the other compound delivering sweetness is 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof. 2,4-dihydroxybenzoic acid is used in the sweetener composition in an amount of, as calculated in the form of the free acid, 0.1 to 4.0 times the amount of aspartame, on a weight basis.

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SWEETENING COMPOSITION COMPRISING ASPARTAME AND 2,4-DIHYDROXYBENZOIC ACID

The invention relates to foodstuffs containing improved sucrose-like tasting sweetener compositions comprising aspartame as the principal source of sweetness and another compound delivering sweetness. In the foodstuffs containing these compositions the sweet taste quality is improved to be closer to that delivered by sucrose. The invention also relates to such improved sucrose-like tasting sweetener compositions, and to an improved method of sweetening edible materials, especially beverages, with aspartame as one of the sources of sweetness. The term "principal source of sweetness" as used herein means that most of the sweetness (in a certain product) originates from the compound.

Aspartame (α-L-aspartyl-L-phenylalanine methyl ester, hereinafter also referred to as APM) is a low calorie dipeptide sweetener with a sweetening power that is about 200x that of sugar (sucrose), and thus belongs to the intense sweeteners most widely used for table-top sweeteners and for sweetening a wide variety of foodstuffs, especially edible products, soft drinks, confectionery, medicines, etc. Aspartame, like other artificial sweeteners, has a sweetening power which is far higher than that of natural sugars, but the taste quality of the sweetness of APM and other intense sweeteners diverges from that of sucrose and for many of the intense sweeteners, especially when used at higher concentrations, more or less strong aftertastes are observed. Sucrose still serves as the standard for

the evaluation of sweetness, owing to the fact that people have become accustomed to it for a long time. Moreover, sucrose imparts a specific mouthfeel in the tasting of sucrose-sweetened products.

Sucrose-like tasting compositions of aspartame with another intense sweetener, namely acesulfame-K (3,4-dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide potassium salt; hereinafter also abbreviated as Ace-K), are known from U.S. Patent No. 4,158,068. They can be used for sweetening many edible materials, including beverages. There is a disadvantage, however, in the fact that under some circumstances the bitter aftertaste of Ace-K can be detected. Thus, choice of ratios of APM vs. Ace-K for practical use remains restricted. Particularly for systems rich in Ace-K the risk exists that some part of the population tasting such compositions will find them to be not only not sucrose-like, but also having less pleasant taste qualities. In addition, even when the starting blend of APM and Ace-K is relatively rich in APM, the composition of the blend may vary over time because, in solution, APM is less stable than Ace-K. Decomposition of APM in the blend (into compounds which do not have the sweetening properties of APM) will result in sweetening blends which become relatively richer in Ace-K, and thus may become more prone to detection of the less pleasant taste qualities mentioned above. Moreover, although APM/Ace-K sweetener blends can provide an adequate sucrose-like taste profile, their sweetness is due to a combination of relatively expensive high intensity sweeteners. Accordingly, there is need for alternative, non-nutritive, sweetener systems which provide sucrose-like taste properties, preferably including the mouthfeel

imparted by sucrose, at lower sweetness equivalence costs.

Sweetness equivalence as used herein and hereinafter is determined as sucrose equivalence; hereinafter, the abbreviation S.E. is used to indicate both sweetness equivalence or sucrose equivalence, interchangeably. Sucrose equivalence (for a given sweetener under given circumstances) is readily known or is easily determined. For example, the amount of a sweetener which is equivalent to 10 wt.% sucrose can be determined by having a panel taste a solution of that sweetener and match its sweetness to a 10 wt.% solution of sucrose. Obviously, sucrose equivalence for other than 10 wt.% sucrose is determined by matching the appropriate sucrose solutions. Sucrose-likeness equally can be determined by taste panel evaluations; in addition to determining the sweetness sensation then also other taste qualities, such as mouthfeel, of the samples tested are matched with sucrose taste qualities at the appropriate concentration.

Surprisingly, the present inventors now have found that foodstuffs containing improved sucrose-like tasting sweetener compositions comprising aspartame as the principal source of sweetness and another compound delivering sweetness are obtained by using as the other sweetness delivering compound 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof, in an amount of, as calculated in the form of the free acid, 0.1 to 4.0 times the amount of aspartame, on a weight basis.

Hereinafter, 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof, will be referred to, unless specifically mentioned otherwise, as 2,4-DHB. Physiologically acceptable salts of 2,4-DHB include acid (i.e. carboxylate) salts

and/or hydroxylate salts, especially sodium, potassium, calcium, magnesium, and ammonium salts and the like. The salts may be preformed or formed in the foodstuff by reaction with typical buffering agents, such as sodium phosphate, potassium citrate, sodium acetate, calcium phosphate (e.g. mono- and tricalcium phosphates) and the like which are also normally employed in foodstuffs to provide the desired pH.

Preferably, 2,4-DHB is used in an amount of, as calculated in the form of the free acid, 0.2 to 2.0 times the amount of aspartame, on a weight basis. Most preferred, 2,4-DHB is used in an amount of 0.25 to 1.5 times the amount of aspartame, on a weight basis.

The concentration of 2,4-DHB, as calculated in the form of the free acid, usually will be chosen so as not to be higher than 1.000 ppm in the foodstuff being sweetened according to the present invention. In particular, the concentration of 2,4-DHB, as calculated in the form of the free acid, will be chosen so as not to be higher than 600 ppm in the foodstuff. Of course, such concentrations may depend on the kind of foodstuff being sweetened according to the present invention. For instance, if the foodstuff is a chewing gum, the concentration of 2,4-DHB (in the chewing gum) may be much higher, e.g. up to 5.000 ppm or higher. It will be clear to the skilled man that such concentrations still fall within the scope of the present invention as claimed herein. If APM and 2,4-DHB are being used as a composition in sugar substitutes, including table-tops etc., then of course the concentration of 2,4-DHB in such sugar substitues may be even higher than in chewing gums. For the purposes of the present patent application, such sugar substitutes, however, are not deemed to be foodstuffs themselves. On the other hand, foodstuffs sweetened with such sugar substitutes or

table-tops as meant here, are foodstuffs according to the invention, and the concentration of 2,4-DHB therein (with normal use of the sugar substitute or table-top) usually will be within the ranges as mentioned above.

Literature about 2,4-DHB so far is ambiguous as to the taste properties thereof. On the one hand it is mentioned, e.g. in U.S. Patent No. 5,232,735, EP-A-0485587 and WO-93/10677, that 2,4-DHB is a sweetness inhibitor and that it is essentially tasteless (in the sense of essentially being neither sweet nor bitter in the initial taste, and of being sweet only at concentrations much higher than 0.05 wt.% or 500 ppm). Based on this information it would be expected that 2,4-DHB would reduce the sweetness of another sweetener. Moreover, it is shown, e.g. in EP-A-0485587, that 2,4-DHB, especially the potassium salt thereof, is very effective as a tastand for reducing the off-taste of KCl or of saccharin (the latter without any suggestion of effects on sweetness). On the other hand US-A-4,871,570 and CA-1,208,966 disclose that 2,4-DHB is sweet, at a sweetness equivalence level of about 2-3 wt.% sucrose solution, when used at a concentration of about 2.000 ppm in beverages, both at pH=7 and at pH=3. Moreover, said sweetness of 2.000 ppm of 2,4-DHB is shown to be additive to that of a 5 wt.% sucrose solution.

Inventors' own recent experiments have established that solutions of 2,4-DHB or its sodium salt in water or in a model soft drink (pH 3.2 buffer solution in various types of buffer systems) are only very faintly sweet at concentrations of 2,4-DHB of about 500 ppm or lower, and that their sweetness at about 2.000 ppm will be in the range of that of 1.3 to 1.7 wt.% sucrose solutions (the lower value observed at a pH of about 3; the higher value in water). This

confirms that DHB may be said to be essentially tasteless at concentrations lower than about 800 ppm.

Thus, while some references disclose 2,4-DHB to be sweet, the overall literature does not disclose or remotely suggest that 2,4-DHB would be an excellent foodstuff sweetener, particularly when employed in combination with a significant sweetening amount of the artificial sweetener aspartame. The latter now surprisingly has been found according to the present invention. This is the more surprising since 2,4-DHB, as is being shown by inventors' comparative experiments described hereinafter, does not give any advantages as a foodstuff sweetener when used in combination with other artificial sweeteners such as Ace-K, alitame or saccharin, or even when used in combination with a blend of APM and Ace-K. On the contrary, sweetness and degree of sucrose-like tasting of such other sweeteners are not influenced at all.

The abovementioned Canadian patent No. 1,208,966 is directed to a wide range of sweetness modifying, i.e. either being a sweetener or a sweetness inhibitor, compounds, which may be added to food products. It is noticed that, according the teaching of said patent, the sweetness modifying agents are added to the food product in an amount in the range of 0.001 to 2 wt.%, most preferably of from 0.1 to 0.5 wt.%. There is no teaching nor suggestion that the surprising effects of using 2,4-DHB at the relatively low concentrations according to the present invention (i.e. preferably lower than 600 ppm or 0.06 wt.% in the foodstuff), and in particular in combination with APM as a sweetener, would be obtainable.

The disclosure of sweetness modifying compounds in said Canadian patent also relates to, inter alia, 3,5-dihydroxybenzoic acid (and its sodium

salt), which are shown to be effective sweetness inhibitors, and to 2,3-dihydroxy benzoic acid (and its sodium salt, together hereinafter referred to as 2,3-DHB). Similarly to 2,4-DHB this latter 2,3-DHB is mentioned to be a sweetener. Its sweetness, at 2000 ppm (pH 3, or 7) is about 2-3% S.E., and is shown to be additive to sweetness of 5% sucrose, as is also disclosed for 2,4-DHB. As demonstrated in the experimental part of the present application, 2,3-DHB, however, surprisingly behaves completely differently from 2,4-DHB when combined at about 500 ppm with APM at about 7.5 % S.E; with 2,3-DHB almost no increase in sweetness is observed. Similar arguments apply with regard to 4-amino-2-hydroxy benzoic acid (4,2-AHB), which compound is also disclosed to be a sweetener (in US-A-4,871,570 and CA-A-1,208,966), though being about 30-50% less sweet than 2,3- or 2,4-DHB. 4,2-AHB is shown to be at least additive to the sweetness of 5% sucrose. There is no teaching on effects in combination with APM.

It is further noticed, that US-A-4,613,512 discloses the use of 3-aminobenzoic acid (and its sodium salt, hereinafter together referred to as 3-AB) as a compound having some sweetness of its own, and being somewhat higher at lower pH. Although it is mentioned for 3-AB that the threshold value for sweetness is at a concentration of about 8-10 mmol/l (i.e. that would be at about 1 wt.%), it is nevertheless disclosed in said U.S. patent that compositions of 3-AB on the one hand, and sucrose or APM on the other, may be used in order to reduce the sucrose or APM content in foodstuffs. Reduction of sucrose or APM is shown to be about 25% at a 3-AB content of 500 ppm, up to about 60% at a 3-AB content of 2.000 ppm. However, there is no teaching nor

suggestion of sucrose-likeness of the 3-AB /APM compositions prepared. According to present inventors' own observations, the degree of potential reduction in sucrose or APM content when using 3-AB, as suggested in said reference, is exaggerated. Further, EP-A-0132444 discloses the use of 3-hydroxybenzoic acid (and its sodium salt, hereinafter together referred to as 3-HB) as a sweetness modifier. 3-HB is said to have some sweetness of its own, with a threshold value for sweetness at a concentration of about 5-7 mmol/l (i.e. that would be at about 0.8 wt.%), and is shown to be used to reduce the content of natural carbohydrate or synthetic intense sweeteners (such as saccharin or APM) in foodstuffs to a substantial extent, even of 50% or more. According to EP-A-0132444 3-HB is being used at a concentration in the range of from about 0.01 to 0.4% (i.e. from about 100 ppm to 4.000 ppm), most preferably from about 0.08 to 0.2% (i.e. from about 800 to 2.000 ppm) by weight of the foodstuff. As can be seen from the examples 3-HB enables similar reduction in sweetener content for APM and saccharin, both in the order of magnitude of about 50%; such similarity in behaviour towards APM and saccharin is, according to present inventors' observations not found for 2,4-DHB. In the aforementioned European patent application there is no teaching nor suggestion of sucrose-likeness of the 3-HB/APM (or /saccharin) compositions prepared. Thus, the abovementioned disclosures on 3-AB, 4,2-AHB and 3-HB do not teach, nor make obvious, the presently claimed inventions relating to 2,4-DHB. They merely show that compounds (structurally quite unrelated to 2,4-DHB) may be used for reduction of sucrose or APM content.

According to the present invention, the concentration of APM in the edible material sweetened,

and the weight ratio of 2,4-DHB vs. APM (i.e. [weight of 2,4-DHB] / [weight of APM]), are chosen so as to obtain the desired level of sweetness of the edible material. For instance, in low pH beverages, the APM concentration in general will be chosen in the range of 250 to 600 ppm, whereas the 2,4-DHB concentration in general will be chosen in the range of 60 to 1.000 ppm, calculated on total weight of the sweetened edible material. For the purpose of this patent application, and for convenience, the concentrations of 2,4-DHB and/or its physiologically acceptable salts are all calculated as if the acid form of 2,4-DHB was used.

For the purposes of this invention, best results are obtained when the 2,4-DHB is employed in a foodstuff in combination with APM, where the APM is present in amounts of above about 2% by wt. of sucrose equivalence, preferably from about 2 to about 11% S.E. by weight, most preferably from about 4 to about 9 % S.E.. That is, the APM preferably should be in the range of from above about 50 ppm to about 750 ppm, most preferably from about 150 to about 520 ppm. It is noticed that the concentrations mentioned here are concentrations of APM at a pH of about 3.2; under neutral pH conditions the APM concentrations necessary for achieving the sucrose equivalence values as indicated are somewhat higher.

Apart from providing a more sucrose-like taste and mouthfeel, use of 2,4-DHB in combination with APM, at the relatively low concentration of 2,4-DHB according to the present invention, does also increase the sweetness of these two compounds synergistically. Significant reduction in use of APM intense sweetener is achieved thereby. This is another advantage of the present invention. It also ensures that risks for

surpassing the acceptable daily intake (ADI-) value for APM become even lower.

The effects of the present invention are also surprising in view of the teachings of the abovementioned WO-93/10677 and EP-A-0485587. The theories about taste interactions, as described in those references, where 2,4-DHB is mentioned as a sweetness inhibitor, are based on findings that:

I. if a molecule is a sweet inhibitor and substantially or essentially tasteless, it not only inhibits or reduces the sweetness of substances, but also inhibits or reduces the bitter taste sensation;

II. if a molecule is a bitter inhibitor and substantially or essentially tasteless, it not only inhibits or reduces the bitter taste of substances, but also inhibits or reduces the sweet taste sensation.

None of these theories is able to predict or suggest the excellent taste properties of the foodstuffs containing sweetener compositions of APM and 2,4-DHB according to the present invention.

The invention also relates to improved sucrose-like tasting sweetener compositions comprising aspartame as the principal source of sweetness and another compound delivering sweetness, wherein the other sweetness delivering compound is 2,4-DHB, in an amount of, as calculated in the form of the free acid, 0.1 to 4.0 times the amount of APM, on a weight basis. Preferably, the amount of 2,4-DHB in the composition, as calculated in the form of the free acid, is 0.2 to 2.0 times the amount of APM, on a weight basis. Most preferred, the amount of 2,4-DHB in the composition is 0.25 to 1.5 times the amount of APM, on a weight basis.

Such sweetening compositions according to the invention are extremely suitable for sweetening of foodstuffs, in particular of beverages. The products sweetened therewith have taste characteristics which give the impression of being sweetened with sucrose. For instance, beverages sweetened according to the present invention provide a syrupy, rounded sweetness profile similar to products sweetened with sucrose, whereas beverages sweetened by APM alone have a more lingering sweetness profile. Although blends of APM and Ace-K can have a more sugar-like sweetness/time profile than APM alone, such blends still lack the sucrose-like mouthfeel of the present invention. The sweetening compositions according to the invention may also be used as sugar substitutes, such as table-top sweeteners.

The invention further relates to an improved method of sweetening edible materials, especially beverages, with aspartame as one of the sources of sweetness. In a first embodiment, the sweetener compositions as used for sweetening foodstuffs according to the present invention may be added as such, that is by adding a mixture of APM and 2,4-DHB or by separately adding the APM and the 2,4-DHB, in a weight ratio and concentrations thereof as indicated below, to foodstuffs which do not contain APM as a source of sweetness and optionally contain any other type of sweetener in an amount which in itself is insufficient to obtain the level of sweetness as desired for the sweetened foodstuff. In a further embodiment, the sweetening compositions as used for sweetening foodstuffs according to the present invention also may be prepared in situ, that is in the foodstuff, when the foodstuff already contains a sweetening amount of APM, by adding thereto, in any

suitable way, an appropriate amount of 2,4-DHB to arrive at the weight ratio of APM and 2,4-DHB and the concentration of 2,4-DHB as indicated below. Of course, also combinations of both embodiments are possible. The ratio between APM and 2,4-DHB (i.e. the free acid or a physiologically acceptable salt thereof) will be such that 2,4-DHB is present in an amount of, as calculated in the form of the free acid, 0.1 to 4.0 times the amount of aspartame, on a weight basis. According to the methods of these embodiments of the invention, that is by the combined use of APM and 2,4-DHB, optimum sucrose-like taste properties will be obtained for products which already contain sucrose or another carbohydrate sweetener and which in addition are being sweetened with APM. Namely, by adding APM alone the sucrose-like taste properties will become less than those obtainable by adding additional; amounts of sucrose or another carbohydrate sweetener for achieving the desired level of increased sweetness. Adding a composition of APM and 2,4-DHB gives an improved result compared to adding APM alone for achieving the desired level of sweetness.

Preferably, 2,4-DHB will be present in an amount of, as calculated in the form of the free acid, 0.2 to 2.0 times the amount of aspartame, on a weight basis. Most preferred, 2,4-DHB will be present in an amount of 0.25 to 1.5 times the amount of aspartame, on a weight basis. The amount of 2,4-DHB, as calculated in the form of the free acid, usually will be chosen so as not to be higher than 1.000 ppm in the foodstuff being sweetened according to the present invention. In particular, the amount of 2,4-DHB, as calculated in the form of the free acid, will be chosen so as not to be higher than 600 ppm in the foodstuff. Of course, such

amounts may depend on the kind of foodstuff being sweetened according to the present invention.

Typical foodstuffs, including pharmaceutical preparations, which may contain the improved sucrose-like tasting sweetener compositions of the present invention are, for example, beverages including soft drinks, carbonated beverages, ready to mix beverages, and the like, processed foods and vegetables, soups, sauces, condiments, breakfast cereals, salad dressings, juices, syrups, desserts, including puddings, gelatin and frozen desserts, like ice creams, sherbets, and icings, confections, toothpaste, mouthwashes, chewing gum, snack foods, intermediate moisture foods (e.g. dog foods) and the like. The improved sucrose-like tasting sweetener compositions also may be used in the production of sugar substitutes, including so-called table-top sweeteners, which may be added to foodstuffs. Concentrations of APM and 2,4-DHB as present in such sugar substitutes and table-tops usually will fall beyond the ranges for those concentrations as mentioned in this patent application for the concentrations in foodstuffs. However, normal use of the sugar substitutes and table-tops will ensure that the concentrations of APM and 2,4-DHB achieved in the food product (e.g. the tea, coffee or other beverage) being sweetened therewith, will fall within the concentration ranges indicated hereinbefore.

Various methods for the production of 2,4-DHB are known. 2,4-DHB, for instance may be produced via a Kolbe reaction, starting from resorcinol, or, also starting from resorcinol, via a Marassé solid-phase carboxylation in the presence of mixed sodium and potassium carboxylates and carbon dioxide as described in US-A-4,996,354.

As can be seen from a publication in Food Technology, October 1996, pages 72-81, 2,4-DHB is admitted on the list of GRAS Flavoring Substances under FEMA No. 3798, for an extensive list of potential uses (except for baked goods). None of these suggested uses, however, discloses or teaches the favorable effects of 2,4-DHB in combination with APM according to the present invention.

In the foodstuffs according to the invention and in the sweetener compositions according to the invention, as well as in the method of sweetening edible materials according to the invention it is preferred that the 2,4-DHB is used in the form of a physiologically acceptable salt thereof. Most preferentially the salt of 2,4-DHB is chosen from the group of sodium or potassium salts thereof. The taste properties and qualities of these salts are better than of 2,4-dihydroxybenzoic acid itself. The free acid has some acidic and slightly astringent characteristics. The sodium and potassium salts are less sour and tasteful and have a cleaner taste overall as compared with the free acid. The desired salts of 2,4-DHB easily can be prepared from 2,4-dihydroxybenzoic acid by neutralizing a concentrated aqueous solution thereof with an appropriate base (for instance sodium hydroxide to prepare 2,4-DHB.Na), crystallizing the formed salt (for instance by cooling) and collecting and drying the crystals after removal of the solvent and appropriate washing.

The effects of the invention can be demonstrated by means of the following Examples and Comparative Examples, which in no way are to be considered to be limiting the invention.

Test methods and chemicals used

In all tests for the determination of sweetness equivalence and of qualitative taste characteristics use was made of formal tasting panels. All sensory assessors were experienced in the general sensory procedures employed, and have extensive experience in participating in sensory tests involving sweeteners. Panel size for the Examples and Comparative Examples mostly was 25.

The taste tests were performed either in water (mineral water, pH = 7), or in a soft drink model systems (pH = 3.2) as described below. The solutions were matched for sweetness to relevant samples from a set of sucrose reference solutions (w/v), each differing by 0.5 or 1% sucrose, as appropriate, and ranging from 0% sucrose to 12% sucrose, as appropiate. All samples were served at a temperature of 20 ± 1°C. Separate test sessions were held for each pH condition. Test solutions, coded with 3 digit random number codes, were presented one at a time, in random order to the panellists. Panellists were also provided with appropriate sucrose solutions marked with the actual sucrose concentration. Panellists were asked to take 3 sips of a test solution, followed by a sip of water (rinsing welll), followed by 3 sips of a sucrose standard, followed by a sip of water, etc., and were encouraged to estimate sweetness to one decimal place. Three minute rest periods were imposed between test solutions. Each panel test was duplicated. Data obtained with the 25 person panel were subjected to statistical analysis to establish the confidence limits of each estimate.

Hydroxybenzoic acid compounds used in the test solutions were obtained, as the free acids, from Aldrich Chemical Company, Gillingham, Dorset, UK. The

purity of 2,4-dihydroxybenzoic acid, as purchased, was about 97%. This impure acid was recrystallized from aqueous ethanol, and harvested crystals were washed with cold water before being dried. Few test also were performed using the hydroxybenzoic acid compound as purchased, and -in fact- no significant differences were observed in taste properties of the beverages prepared from the impure or purified product, even though the recrystallized product itself was found to be more acidic (but generally cleaner) in taste.

Physiologically acceptable salts, in particular sodium salts, of the hydroxybenzoic acid compounds used were prepared from the hydroxybenzoic acid compounds by dissolving appropriate amounts thereof in about 1M aqueous sodium hydroxide, while stirring at 50 °C and adjusting the pH of the solution to 6.0, then adding an about equal volume of ethanol (96%) and cooling overnight in the refrigerator at about 4 °C, followed by evaporation under vacuum to obtain a slurry of the sodium salt from which this salt can be recovered by solid-liquid separation on a Büchner funnel with washing using ice-cold water, and drying of the crystals.

Soft drink model systems

The composition of the soft drink model systems (A. Cola; B. Lemon/lime carbonate) was as follows, each time adding 1 part of the syrup (of table I) to 5.5 parts of carbonated water:

Table I:

Cola (A)		Lemon/lime (B)	
Formulation	**wt.%**	**Formulation**	**wt.%**
Cola Base (AK70108B)*	1.6250	Citric acid	0.9100
Cola Flavour (AK70108A)*	0.6500	Lemon flavour (17.42.6240)**	0.300
Phosphoric acid (85% solution)	0.5005	Lime flavour (17.42.5408)**	0.300
Sodium citrate	0.2340	Sodium citrate	0.2600
Sodium benzoate	0.0975	Sodium benzoate	0.0975
Sweetener(s)	As required	Sweetener(s)	As required
Water to	100.0000	Water to	100.0000

* Perlarom

** IFF

Sweetness dose response for 2,4-DHB

Sweetness dose response curves were determined by panel method for 2,4-DHB (both as free acid and in the form of its sodium salt; the concentration of the latter being calculated in the form of the free acid) in a buffer solution (pH = 3.2; 0.14% citric acid, 0.04% sodium citrate) and in mineral water (pH = 7). The results (with no differences for the free acid and the sodium salt) are summarized in table II:

Table II

dose response for 2,4-DHB	at pH = 3.2	at pH = 7.0
concentration (ppm)	S.E. (%)	S.E. (%)
0	0.36	0.17
125	0.50	0.22
250	0.62	0.41
375	0.87	0.59
500	0.92	0.76
1000	1.36	1.35
1500	1.70	1.67
2000	1.72	1.82

Example 1: Sweetness of various APM/2,4-DHB compositions (small panel tests)

Various compositions of APM and 2,4-DHB in a pH = 3.2 buffer of citric acid / sodium citrate (at APM concentrations ranging from 0 to 600 ppm, and 2,4-DHB-concentrations of respectively 250 and 500 ppm) were tasted for sweetness equivalence, and compared with standard APM solutions at that pH without 2,4-DHB. The results are summarized in table III.

Table III

conc. APM (ppm)	average S.E. (%)		
	0 ppm 2,4-DHB	250 ppm 2,4-DHB	500 ppm DHB
0	0.36	0.62	0.92
100	2.34	3.60	4.23
200	4.75	5.52	6.43
300	6.45	7.26	7.89
400	7.21	8.60	9.26
500	9.05	9.86	10.72
600	9.50	10.58	11.25

It can be seen that the S.E. of the compositions is higher than would be expected by mere addition of the S.E.'s of the individual components at their respective concentrations. The compositions have excellent sucrose-like characteristics.

It was further established (through tests of compositions of 400 ppm of APM together with either 250 or 500 ppm of 2,4-DHB in 0.073% phosphoric acid buffer and in 0.1%/0.02% malic acid/malate buffer at pH = 3.2), that the results as summarized in Table II are more or less independent of the buffer system used.

<u>Examples 2-4: APM/2,4-DHB in soft drink model systems, and Comparative Examples A-D</u>

Cola and lemon/lime soft drink model systems having a syrup composition as shown above, with added sweetener as shown below in amounts as present in the diluted soft drink, were prepared and subjected to sensory evaluations by the panellists. The sweetener composition of the soft drinks as tested, as well as the observed S.E. values, are summarized in table IV below.

Table IV

Example or Comp.Ex.	Soft drink	Sweetener type	Sweetener ppm (wt.%)	S.E.	
C.Ex. A	Cola	sucrose	(11.0)	about 11	sucrose-like (by nature)
C.Ex. B	Cola	APM	520	about 9	less sucrose-like; not syrupy and rounded
Ex.2	Cola	APM 2,4-DHB	350 500	about 9	extremely sucrose-like; syrupy and rounded profile
Ex.3	Cola	APM 2,4-DHB	430 215	slightly sweeter than Ex.2	extremely sucrose-like; syrupy and rounded profile

Example or Comp.Ex.	Soft drink	Sweetener type	Sweetener ppm (wt.%)	S.E.	
C.Ex. C	Lemon/lime	sucrose	(9.0)		sucrose-like (by nature)
C.Ex. D	Lemon/lime	APM	500	about 9	less sucrose-like; not syrupy and rounded
Ex.4	Lemon/lime	APM 2,4-DHB	375 500	about 9	extremely sucrose-like; syrupy and rounded

Example 5 and Comparative Examples E-H: Comparison of APM / 2,4-DHB compositions with compositions of 2,4-DHB and other sweeteners or sweetener blends.

Sweetness evaluation tests were performed, in a buffer system of pH 3.2 (0.14% citric acid, 0.04% sodium citrate) for compositions containing 2,4-DHB and another sweetener as shown in table V. The S.E. values observed clearly indicate the surprising effects on sweetness for the compositions according to the invention as compared with those where APM is replaced by another sweetener.

Table V:

Comparative Examples E-H / Example 5

	other intense sweetener		average S.E. (%) 0 ppm 2,4-DHB	250 ppm 2,4-DHB (S.E.=0.41%)	500 ppm 2,4-DHB (S.E.=0.71%)
	type	ppm			
E.	alitame	30	7.74	-	7.93
F.	Ace-K	a) 200 b) 1000	3.66 7.19	3.68 7.20	3.56 6.87
G.	APM/Ace-K 1:1	140+140	8.46	8.60	8.52
H.	saccharinate	a) 100 b) 650	3.76 7.24	3.87 7.10	3.68 7.35
5	APM	a) 300 b) 500	6.45 9.05	7.26 9.86	7.89 10.35

Example 6 and Comparative Examples I-L: Comparison of APM / 2,4-DHB compositions with compositions of APM and other sweetness modifying compounds.

Sweetness evaluation tests were performed, in a buffer system of pH 3.2 (0.14% citric acid, 0.04% sodium citrate) for compositions containing APM and another sweetness modifying compound as shown in table VI. The S.E. values observed clearly indicate the surprising effects on sweetness for the compositions according to the invention as compared with those where 2,4-DHB is replaced by another sweetness modifying compound.

Table VI:

		average S.E. (%)		
	sweetness modifying compound (SMC) (Comp. Expls I-L, Expl.6)	400 ppm APM 0 ppm SMC	0 ppm APM 500 ppm SMC	400 ppm APM + 500 ppm SMC
I.	2,3-DHB slightly astringent	7.49	0.58	8.03
J.	2,5-DHB	7.49	0.38	7.32
K.	3,5-DHB	7.49	0.56	6.74
L.	4-amino-2-hydroxy benzoic acid	7.49	0.58	8.26
6	2,4-DHB	7.49	0.92	9.28

CLAIMS

1. Foodstuff containing improved sucrose-like tasting sweetener compositions comprising aspartame as the principal source of sweetness and another compound delivering sweetness, characterized in that the foodstuff contains as the other sweetness delivering compound 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof, in an amount of, as calculated in the form of the free acid, 0.1 to 4.0 times the amount of aspartame, on a weight basis.
2. Foodstuff according to claim 1, characterized in that it contains 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof in an amount of, as calculated in the form of the free acid, 0.2 to 2.0 times the amount of aspartame, on a weight basis.
3. Foodstuff according to claim 1 or 2, characterized in that it contains 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof in an amount of, as calculated in the form of the free acid, 0.25 to 1.5 times the amount of aspartame, on a weight basis.
4. Foodstuff according to any of claims 1-3, characterized in that the concentration of 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof, as calculated in the form of the free acid, in the foodstuff is not higher than 1.000 ppm.

5. Foodstuff according to claim 4, characterized in that the concentration of 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof, as calculated in the form of the free acid, in the foodstuff is not higher than 600 ppm.

6. Foodstuff according to any of claims 1-5, characterized in that the aspartame in the foodstuff is present in an amount of from about 2 to about 12% by wt. of sucrose equivalence.

7. Improved sucrose-like tasting sweetener composition comprising aspartame as the principal source of sweetness and another compound delivering sweetness, characterized in that the compositition contains as the other sweetness delivering compound 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof in an amount of, as calculated in the form of the free acid, 0.1 to 4.0, in particular 0.2 to 2.0, most particularly 0.25 to 1.5, times the amount of aspartame, on a weight basis.

8. Improved method of sweetening edible materials, especially beverages, with aspartame as one of the sources of sweetness, characterized in that a mixture of aspartame and 2,4-dihydroxybenzoic acid or a physiologically acceptable salt thereof is added together or separately, to foodstuffs which do not contain APM as a source of sweetness and optionally contain any other type of sweetener in an amount which in itself is insufficient to obtain the level of sweetness to be achieved in the sweetened foodstuff, in a weight ratio and concentrations thereof such that the amount of

2,4-dihydroxybenzoic acid or the physiologically acceptable salt thereof, as calculated in the form of the free acid, is from 0.1 to 4.0 times the amount of aspartame, on a weight basis, and its concentration is not higher than 1.000 ppm in the foodstuff.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/NL 98/00528

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A23L1/236

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 678 026 A (ARIYOSHI YASUO ET AL) 18 July 1972	1-3,7
A	see examples 1,5	4-6,8
X	EP 0 333 036 A (MITSUI TOATSU CHEMICALS) 20 September 1989 see Comparative Example 1	1-3,8

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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1

INTERNATIONAL SEARCH REPORT

Information on patent family members

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